

## 2-(4-Methoxy-2-methylanilino)-1,2-diphenylethanone

Hakan Arslan,<sup>a,b\*</sup> Oztekin Algul,<sup>c</sup> Selin Zirek,<sup>c</sup> Ozden Tari,<sup>c</sup> Aydin Demircan<sup>d</sup> and Kenneth I. Hardcastle<sup>a</sup><sup>a</sup>Department of Chemistry, Emory University, Atlanta, GA 30322, USA, <sup>b</sup>Department of Chemistry, Faculty of Arts and Science, Mersin University, Mersin, TR 33343, Turkey, <sup>c</sup>Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Mersin University, Mersin, TR 33169, Turkey, and <sup>d</sup>Department of Chemistry, Faculty of Arts and Sciences, Nigde University, Nigde, TR 51240, Turkey

Correspondence e-mail: hakan.arslan.acad@gmail.com

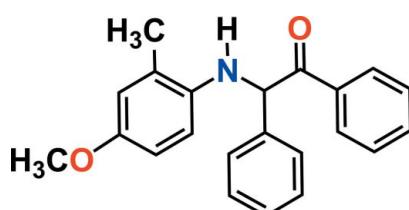
Received 26 April 2011; accepted 27 April 2011

Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.042;  $wR$  factor = 0.112; data-to-parameter ratio = 18.2.

The title compound,  $\text{C}_{22}\text{H}_{21}\text{NO}_2$ , was synthesized from 4-methoxy-2-methylaniline and 2-hydroxy-1,2-diphenylethanone. In the title compound, the  $\text{C}-\text{C}-\text{C}-\text{N}-\text{C}$  backbone adopts an all-*trans* conformation. The crystal structure is stabilized by weak intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen-bond interactions.

## Related literature

For the synthesis and similar structures, see: Au & Tafeenko (1986, 1987); Batsanov *et al.* (2006). For general background to these structures, see: Batsanov *et al.* (2006); Abdulla *et al.* (1985). For bond-length data, see: Allen *et al.* (1987). For geometrical analysis, see: Bruno *et al.* (2002).



## Experimental

## Crystal data

$\text{C}_{22}\text{H}_{21}\text{NO}_2$   
 $M_r = 331.40$   
Monoclinic,  $P2_1/c$

$a = 12.570$  (12) Å  
 $b = 8.009$  (8) Å  
 $c = 18.091$  (17) Å

$\beta = 100.544$  (15)°  
 $V = 1791$  (3) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation

$\mu = 0.08$  mm<sup>-1</sup>  
 $T = 173$  K  
 $0.21 \times 0.19 \times 0.15$  mm

## Data collection

Bruker SMART CCD area-detector diffractometer  
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)  
 $T_{\min} = 0.984$ ,  $T_{\max} = 0.988$

28799 measured reflections  
4122 independent reflections  
2935 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.055$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.112$   
 $S = 1.05$   
4122 reflections

226 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.27$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.25$  e Å<sup>-3</sup>

**Table 1**  
Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
C11—H11A···O1 <sup>i</sup>	0.95	2.48	3.352 (4)	153

Symmetry code: (i)  $x, y + 1, z$ .

Data collection: *SMART* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008), *OLEX2*, *publCIF* (Westrip, 2010) and *Mercury* (Macrae *et al.*, 2006).

This work was supported by the Mersin University Research Fund [Project No. BAP-SBE FK (SZ) 2008–8 YL].

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5030).

## References

Abdulla, R. F., Boyd, D. B., Jones, N. D. & Swartzendruber, J. K. (1985). *J. Org. Chem.* **50**, 3502–3505.  
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.  
Au, O. & Tafeenko, V. (1986). *Rev. Cubana Quim.* **2**, 65–74.  
Au, O. & Tafeenko, V. (1987). *Rev. Cubana Quim.* **3**, 79–86.  
Batsanov, A. S., Goeta, A. E., Howard, J. A. K., Soto, B. & Au-Alvarez, O. (2006). *Acta Cryst.* **C62**, o304–o306.  
Bruker (2008). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.  
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.  
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.  
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

## **supplementary materials**

**2-(4-Methoxy-2-methylanilino)-1,2-diphenylethanone****H. Arslan, O. Algul, S. Zirek, O. Tari, A. Demircan and K. I. Hardcastle****Comment**

A few 1-arylanilinoethanone derivatives have been structurally characterized because of their importance in synthesis or because of their interesting charge-transfer properties (Abdulla *et al.*, 1985). We found only four structurally similar compounds (Au & Tafeenko, 1987; Au & Tafeenko, 1986; Batsanov *et al.*, 2006) in the Cambridge Structural Database (CSD CONQUEST 1.11, B4; Bruno *et al.*, 2002).

Compound **I** was synthesized by the HCl acid-catalyzed reaction of a benzoin derivative to 4-methoxy-2-methylaniline (Scheme 1) resulting in the title compound, 2-((4-methoxy-2-methylphenyl)amino)-1,2-diphenylethanone, **I**, Figure 1, its structure was determined by X-ray crystallography.

The molecular structure of the title compound contains two phenyl rings and one substituted aniline ring connected by a  $-\text{C}(\text{O})-\text{C}-$  linker. The dihedral angle between the two phenyl rings is  $87.78(7)^\circ$  and the dihedral angle between the substitute aniline and phenyl rings are  $55.30(7)$  and  $84.57(7)^\circ$ , respectively. In addition, in the title compound, the  $\text{C}2-\text{C}1-\text{C}8-\text{N}1-\text{C}15$  backbone adopts an all-*trans* conformation (Au & Tafeenko, 1987; Au & Tafeenko, 1986; Batsanov *et al.*, 2006).

The  $\text{C}-\text{N}$  bond lengths  $\text{C}8-\text{N}1$  and  $\text{C}15-\text{N}1$  are shorter than the normal  $\text{C}-\text{N}$  single-bond length of about  $1.48\text{\AA}$ . The shortening of these  $\text{C}-\text{N}$  bonds reveals the effects of some conjugation in this part of the molecule. All other bond lengths fall within the expected ranges (Allen *et al.*, 1987).

Intramolecular hydrogen bonding  $\text{N}1-\text{H}1\text{A}\cdots\text{O}1$  with  $\text{N}-\text{H} 0.88\text{\AA}$ ,  $\text{H}\cdots\text{O} 2.22\text{\AA}$ ,  $\text{N}-\text{H}\cdots\text{O} 107^\circ$  results in the formation of a five membered ring in the  $\text{O}1-\text{C}1-\text{C}8-\text{N}1-\text{H}1\text{A}$  plane. The crystal packing is dominated by weak intermolecular  $\text{C}11-\text{H}11\text{A}\cdots\text{O}1$  ( $x, 1+y, z$ ) hydrogen bonds, with  $\text{H}\cdots\text{O} = 2.48\text{\AA}$  and a  $\text{C}-\text{H}\cdots\text{O}$  angle of  $153^\circ$  (Figure 2).

**Experimental**

A mixture of 4-methoxy-2-methylaniline (15 mmol), 2-hydroxy-1,2-diphenylethanone (5 mmol) and 1 ml conc. HCl in 20 ml of ethanol were refluxed for 5 h (Figure 3). After reaction was complete, the mixture was allowed to cool to room temperature, poured into cold water (20 ml) and finally extracted with  $\text{CH}_2\text{Cl}_2$  (3x15 ml). The organic layer was dried over magnesium sulfate and the solvent removed under reduced pressure to yield a crude product that was purified by recrystallization in ethyl acetate. 2-((4-methoxy-2-methylphenyl)amino)-1,2-diphenylethanone: Yield: 1.20 g, 46%. *M.p.*: 110–112 °C.  $^1\text{H}$  NMR (DMSO-*d*<sub>6</sub>)  $\delta$ : 8.18–8.16 (d, 2H, Ar—H (C3, C7)), 7.62 (t, 1H, Ar—H (C5)), 7.56–7.49 (m, 4H, Ar—H (C4, C6, C11, C13)), 7.30–7.26 (m, 2H, Ar—H (C10, C14)), 7.16 (t, 1H, Ar—H (C12)), 6.70–6.68 (d,  $J=4.8$  Hz, 1H, Ar—H (C19)), 6.67 (s, 1H, Ar—H (C17)), 6.57–6.55 (d,  $J=8$  Hz, 1H, Ar—H (C20)), 6.46–6.44 (d,  $J=8$  Hz, 1H, Ar—H (C8)), 5.19–5.17 (d,  $J=8$  Hz, 1H, NH), 3.60 (s, 3H, OCH<sub>3</sub>), 2.22 (s, 3H, CH<sub>3</sub>).  $^{13}\text{C}$  NMR (400 MHz, p.p.m.)  $\delta$ : 17.5 (CH<sub>3</sub>), 55.1 (OCH<sub>3</sub>), 61.5 (C8), 111.2 (C19), 112.5 (C20), 116.4 (C17), 123.9 (C16), 127.6, 128.1, 128.6, 128.7, 128.8 (C), 133.7

## supplementary materials

---

(C5), 134.7 (C2), 138.1 (C9), 138.3 (C15), 151.1 (C18), 197.6 (C9). Anal. Calc. for  $C_{22}H_{21}NO_2$ : C, 79.73; H, 6.39; N, 4.23%. Found: C, 79.70; H, 6.21; N, 4.19%.

### Refinement

H atom positions were clearly derived from difference Fourier maps and refined using a riding model, fixing the bond lengths at 0.98 and 0.95 Å for  $CH_3$  and  $CH$ (aromatic), respectively. The displacement parameters of the H atoms were constrained with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}$  (C) or  $1.5U_{\text{eq}}$  (methyl C).

### Figures

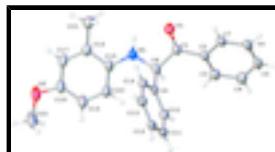


Fig. 1. The molecular structure of (I), showing ellipsoids at the 50% probability level.



Fig. 2. The molecular packing of (I). The hydrogen bonds are shown as dashed lines.

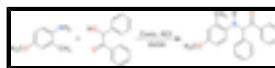


Fig. 3. Synthesis of the title compound.

### 2-(4-Methoxy-2-methylanilino)-1,2-diphenylethanone

#### Crystal data

$C_{22}H_{21}NO_2$	$F(000) = 704$
$M_r = 331.40$	$D_x = 1.229 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 4852 reflections
$a = 12.570 (12) \text{ \AA}$	$\theta = 2.3\text{--}24.5^\circ$
$b = 8.009 (8) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 18.091 (17) \text{ \AA}$	$T = 173 \text{ K}$
$\beta = 100.544 (15)^\circ$	Block, yellow
$V = 1791 (3) \text{ \AA}^3$	$0.21 \times 0.19 \times 0.15 \text{ mm}$
$Z = 4$	

#### Data collection

Bruker SMART CCD area-detector	4122 independent reflections
diffractometer	
Radiation source: fine-focus sealed tube	2935 reflections with $I > 2\sigma(I)$
graphite	$R_{\text{int}} = 0.055$
$\varphi$ and $\omega$ scans	$\theta_{\text{max}} = 27.5^\circ, \theta_{\text{min}} = 1.7^\circ$
Absorption correction: multi-scan	$h = -16 \rightarrow 16$

(SADABS; Bruker, 2008)

$T_{\min} = 0.984$ ,  $T_{\max} = 0.988$   
28799 measured reflections

$k = -10 \rightarrow 10$   
 $l = -23 \rightarrow 23$

### Refinement

Refinement on  $F^2$

Primary atom site location: structure-invariant direct methods

Least-squares matrix: full

Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.042$

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.112$

H-atom parameters constrained

$S = 1.05$

$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.4229P]$

where  $P = (F_o^2 + 2F_c^2)/3$

4122 reflections

$(\Delta/\sigma)_{\max} = 0.004$

226 parameters

$\Delta\rho_{\max} = 0.27 \text{ e \AA}^{-3}$

0 restraints

$\Delta\rho_{\min} = -0.25 \text{ e \AA}^{-3}$

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.70413 (11)	0.71666 (18)	0.13255 (8)	0.0336 (3)
C2	0.65511 (11)	0.79413 (17)	0.05905 (7)	0.0314 (3)
C3	0.70228 (12)	0.92642 (19)	0.02686 (8)	0.0380 (3)
H3A	0.7667	0.9764	0.0533	0.046*
C4	0.65510 (14)	0.9856 (2)	-0.04399 (8)	0.0453 (4)
H4A	0.6879	1.0748	-0.0662	0.054*
C5	0.56034 (14)	0.9144 (2)	-0.08206 (8)	0.0436 (4)
H5A	0.5286	0.9546	-0.1305	0.052*
C6	0.51179 (13)	0.7853 (2)	-0.05009 (8)	0.0420 (4)
H6A	0.4460	0.7385	-0.0761	0.050*
C7	0.55897 (12)	0.72392 (18)	0.01989 (8)	0.0361 (3)
H7A	0.5260	0.6339	0.0414	0.043*
C8	0.78651 (11)	0.81396 (18)	0.19059 (8)	0.0339 (3)
H8A	0.8385	0.8744	0.1644	0.041*
C9	0.72052 (11)	0.94044 (17)	0.22728 (7)	0.0297 (3)

## supplementary materials

---

C10	0.72302 (12)	1.11045 (18)	0.21167 (8)	0.0370 (3)
H10A	0.7685	1.1508	0.1790	0.044*
C11	0.65930 (13)	1.22179 (18)	0.24361 (9)	0.0421 (4)
H11A	0.6615	1.3376	0.2327	0.051*
C12	0.59250 (12)	1.16418 (19)	0.29135 (8)	0.0396 (4)
H12A	0.5489	1.2403	0.3129	0.048*
C13	0.58976 (12)	0.99566 (19)	0.30736 (8)	0.0378 (3)
H13A	0.5441	0.9557	0.3399	0.045*
C14	0.65372 (11)	0.88458 (18)	0.27584 (8)	0.0334 (3)
H14A	0.6519	0.7691	0.2875	0.040*
C15	0.91952 (11)	0.72896 (19)	0.30593 (8)	0.0330 (3)
C16	0.95263 (11)	0.59902 (19)	0.35825 (8)	0.0346 (3)
C17	1.02943 (11)	0.6351 (2)	0.42183 (8)	0.0393 (4)
H17A	1.0524	0.5485	0.4570	0.047*
C18	1.07393 (12)	0.7936 (2)	0.43580 (8)	0.0412 (4)
C19	1.04216 (12)	0.9204 (2)	0.38448 (8)	0.0409 (4)
H19A	1.0725	1.0289	0.3929	0.049*
C20	0.96492 (11)	0.88712 (19)	0.32004 (8)	0.0375 (3)
H20A	0.9429	0.9745	0.2851	0.045*
C21	0.90500 (13)	0.4262 (2)	0.34587 (9)	0.0442 (4)
H21A	0.9374	0.3535	0.3875	0.066*
H21B	0.9201	0.3810	0.2985	0.066*
H21C	0.8266	0.4318	0.3435	0.066*
C22	1.19345 (16)	0.9696 (3)	0.52047 (11)	0.0690 (6)
H22A	1.2440	0.9640	0.5686	0.103*
H22B	1.1358	1.0496	0.5244	0.103*
H22C	1.2322	1.0058	0.4810	0.103*
N1	0.84386 (10)	0.69119 (16)	0.24129 (7)	0.0412 (3)
H1A	0.8302	0.5852	0.2308	0.049*
O1	0.67637 (9)	0.57676 (13)	0.14827 (6)	0.0477 (3)
O2	1.14758 (10)	0.80923 (17)	0.50230 (6)	0.0612 (4)

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0331 (7)	0.0372 (8)	0.0294 (7)	-0.0008 (6)	0.0026 (6)	-0.0002 (6)
C2	0.0323 (7)	0.0345 (8)	0.0264 (7)	0.0029 (6)	0.0024 (6)	-0.0041 (6)
C3	0.0388 (8)	0.0420 (8)	0.0318 (8)	-0.0004 (7)	0.0030 (6)	-0.0009 (6)
C4	0.0585 (10)	0.0449 (9)	0.0338 (8)	0.0049 (8)	0.0114 (7)	0.0045 (7)
C5	0.0558 (10)	0.0458 (9)	0.0256 (7)	0.0141 (8)	-0.0022 (7)	-0.0025 (6)
C6	0.0435 (9)	0.0450 (9)	0.0326 (8)	0.0067 (7)	-0.0062 (7)	-0.0089 (7)
C7	0.0386 (8)	0.0360 (8)	0.0318 (7)	0.0014 (6)	0.0013 (6)	-0.0056 (6)
C8	0.0314 (7)	0.0377 (8)	0.0299 (7)	-0.0001 (6)	-0.0013 (6)	0.0027 (6)
C9	0.0279 (7)	0.0321 (7)	0.0252 (7)	-0.0033 (6)	-0.0055 (5)	0.0023 (5)
C10	0.0399 (8)	0.0347 (8)	0.0331 (7)	-0.0082 (6)	-0.0024 (6)	0.0067 (6)
C11	0.0503 (9)	0.0265 (7)	0.0418 (8)	-0.0024 (7)	-0.0121 (7)	0.0012 (6)
C12	0.0401 (8)	0.0371 (8)	0.0362 (8)	0.0056 (7)	-0.0076 (7)	-0.0060 (6)
C13	0.0381 (8)	0.0399 (8)	0.0337 (7)	-0.0025 (7)	0.0023 (6)	-0.0008 (6)

C14	0.0373 (8)	0.0288 (7)	0.0314 (7)	-0.0032 (6)	-0.0006 (6)	0.0029 (6)
C15	0.0260 (7)	0.0444 (8)	0.0281 (7)	0.0042 (6)	0.0033 (5)	0.0017 (6)
C16	0.0279 (7)	0.0453 (9)	0.0315 (7)	0.0023 (6)	0.0084 (6)	0.0050 (6)
C17	0.0302 (7)	0.0542 (10)	0.0333 (8)	0.0001 (7)	0.0047 (6)	0.0151 (7)
C18	0.0297 (8)	0.0616 (10)	0.0295 (7)	-0.0071 (7)	-0.0016 (6)	0.0101 (7)
C19	0.0351 (8)	0.0499 (9)	0.0356 (8)	-0.0089 (7)	0.0011 (6)	0.0052 (7)
C20	0.0337 (8)	0.0445 (9)	0.0323 (7)	0.0018 (7)	0.0007 (6)	0.0082 (6)
C21	0.0437 (9)	0.0476 (9)	0.0407 (8)	-0.0011 (7)	0.0062 (7)	0.0079 (7)
C22	0.0614 (12)	0.0892 (15)	0.0463 (10)	-0.0308 (11)	-0.0170 (9)	0.0086 (10)
N1	0.0441 (7)	0.0359 (7)	0.0371 (7)	0.0081 (6)	-0.0094 (6)	-0.0035 (5)
O1	0.0572 (7)	0.0424 (6)	0.0380 (6)	-0.0124 (5)	-0.0057 (5)	0.0068 (5)
O2	0.0565 (7)	0.0759 (9)	0.0411 (6)	-0.0243 (7)	-0.0179 (6)	0.0195 (6)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C1—O1	1.2226 (19)	C12—H12A	0.9500
C1—C2	1.494 (2)	C13—C14	1.390 (2)
C1—C8	1.544 (2)	C13—H13A	0.9500
C2—C3	1.393 (2)	C14—H14A	0.9500
C2—C7	1.402 (2)	C15—C20	1.394 (2)
C3—C4	1.393 (2)	C15—N1	1.399 (2)
C3—H3A	0.9500	C15—C16	1.417 (2)
C4—C5	1.385 (2)	C16—C17	1.390 (2)
C4—H4A	0.9500	C16—C21	1.509 (2)
C5—C6	1.380 (2)	C17—C18	1.391 (2)
C5—H5A	0.9500	C17—H17A	0.9500
C6—C7	1.386 (2)	C18—O2	1.383 (2)
C6—H6A	0.9500	C18—C19	1.385 (2)
C7—H7A	0.9500	C19—C20	1.399 (2)
C8—N1	1.4443 (19)	C19—H19A	0.9500
C8—C9	1.535 (2)	C20—H20A	0.9500
C8—H8A	1.0000	C21—H21A	0.9800
C9—C10	1.392 (2)	C21—H21B	0.9800
C9—C14	1.395 (2)	C21—H21C	0.9800
C10—C11	1.393 (2)	C22—O2	1.422 (2)
C10—H10A	0.9500	C22—H22A	0.9800
C11—C12	1.389 (2)	C22—H22B	0.9800
C11—H11A	0.9500	C22—H22C	0.9800
C12—C13	1.382 (2)	N1—H1A	0.8800
O1—C1—C2	119.92 (13)	C12—C13—H13A	120.0
O1—C1—C8	119.27 (13)	C14—C13—H13A	120.0
C2—C1—C8	120.77 (13)	C13—C14—C9	120.87 (14)
C3—C2—C7	119.18 (14)	C13—C14—H14A	119.6
C3—C2—C1	123.38 (13)	C9—C14—H14A	119.6
C7—C2—C1	117.39 (13)	C20—C15—N1	122.87 (13)
C2—C3—C4	120.09 (15)	C20—C15—C16	119.00 (14)
C2—C3—H3A	120.0	N1—C15—C16	118.12 (14)
C4—C3—H3A	120.0	C17—C16—C15	118.33 (15)
C5—C4—C3	119.99 (16)	C17—C16—C21	120.70 (13)

## supplementary materials

---

C5—C4—H4A	120.0	C15—C16—C21	120.97 (14)
C3—C4—H4A	120.0	C16—C17—C18	122.38 (14)
C6—C5—C4	120.41 (15)	C16—C17—H17A	118.8
C6—C5—H5A	119.8	C18—C17—H17A	118.8
C4—C5—H5A	119.8	O2—C18—C19	125.58 (15)
C5—C6—C7	120.02 (15)	O2—C18—C17	115.06 (13)
C5—C6—H6A	120.0	C19—C18—C17	119.35 (14)
C7—C6—H6A	120.0	C18—C19—C20	119.34 (15)
C6—C7—C2	120.30 (15)	C18—C19—H19A	120.3
C6—C7—H7A	119.9	C20—C19—H19A	120.3
C2—C7—H7A	119.9	C15—C20—C19	121.61 (14)
N1—C8—C9	114.91 (13)	C15—C20—H20A	119.2
N1—C8—C1	106.44 (13)	C19—C20—H20A	119.2
C9—C8—C1	106.20 (12)	C16—C21—H21A	109.5
N1—C8—H8A	109.7	C16—C21—H21B	109.5
C9—C8—H8A	109.7	H21A—C21—H21B	109.5
C1—C8—H8A	109.7	C16—C21—H21C	109.5
C10—C9—C14	118.68 (13)	H21A—C21—H21C	109.5
C10—C9—C8	121.58 (13)	H21B—C21—H21C	109.5
C14—C9—C8	119.71 (13)	O2—C22—H22A	109.5
C9—C10—C11	120.40 (15)	O2—C22—H22B	109.5
C9—C10—H10A	119.8	H22A—C22—H22B	109.5
C11—C10—H10A	119.8	O2—C22—H22C	109.5
C12—C11—C10	120.28 (15)	H22A—C22—H22C	109.5
C12—C11—H11A	119.9	H22B—C22—H22C	109.5
C10—C11—H11A	119.9	C15—N1—C8	124.59 (13)
C13—C12—C11	119.71 (14)	C15—N1—H1A	117.7
C13—C12—H12A	120.1	C8—N1—H1A	117.7
C11—C12—H12A	120.1	C18—O2—C22	117.49 (13)
C12—C13—C14	120.05 (15)		
O1—C1—C2—C3	−160.36 (14)	C11—C12—C13—C14	0.1 (2)
C8—C1—C2—C3	22.1 (2)	C12—C13—C14—C9	−0.6 (2)
O1—C1—C2—C7	17.0 (2)	C10—C9—C14—C13	0.8 (2)
C8—C1—C2—C7	−160.60 (12)	C8—C9—C14—C13	−177.33 (12)
C7—C2—C3—C4	−1.2 (2)	C20—C15—C16—C17	0.0 (2)
C1—C2—C3—C4	176.15 (14)	N1—C15—C16—C17	178.73 (13)
C2—C3—C4—C5	0.9 (2)	C20—C15—C16—C21	179.37 (13)
C3—C4—C5—C6	0.3 (2)	N1—C15—C16—C21	−1.9 (2)
C4—C5—C6—C7	−1.3 (2)	C15—C16—C17—C18	0.4 (2)
C5—C6—C7—C2	1.0 (2)	C21—C16—C17—C18	−178.96 (14)
C3—C2—C7—C6	0.2 (2)	C16—C17—C18—O2	178.75 (13)
C1—C2—C7—C6	−177.25 (13)	C16—C17—C18—C19	−0.8 (2)
O1—C1—C8—N1	21.30 (18)	O2—C18—C19—C20	−178.70 (14)
C2—C1—C8—N1	−161.10 (12)	C17—C18—C19—C20	0.8 (2)
O1—C1—C8—C9	−101.58 (16)	N1—C15—C20—C19	−178.65 (14)
C2—C1—C8—C9	76.02 (16)	C16—C15—C20—C19	0.0 (2)
N1—C8—C9—C10	135.07 (14)	C18—C19—C20—C15	−0.4 (2)
C1—C8—C9—C10	−107.56 (15)	C20—C15—N1—C8	−14.1 (2)
N1—C8—C9—C14	−46.83 (17)	C16—C15—N1—C8	167.24 (13)

C1—C8—C9—C14	70.54 (15)	C9—C8—N1—C15	−57.18 (19)
C14—C9—C10—C11	−0.5 (2)	C1—C8—N1—C15	−174.41 (13)
C8—C9—C10—C11	177.64 (12)	C19—C18—O2—C22	1.2 (2)
C9—C10—C11—C12	−0.1 (2)	C17—C18—O2—C22	−178.33 (16)
C10—C11—C12—C13	0.3 (2)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···O1	0.88	2.22	2.609 (3)	107
C11—H11A···O1 <sup>i</sup>	0.95	2.48	3.352 (4)	153

Symmetry codes: (i)  $x, y+1, z$ .

## supplementary materials

---

Fig. 1

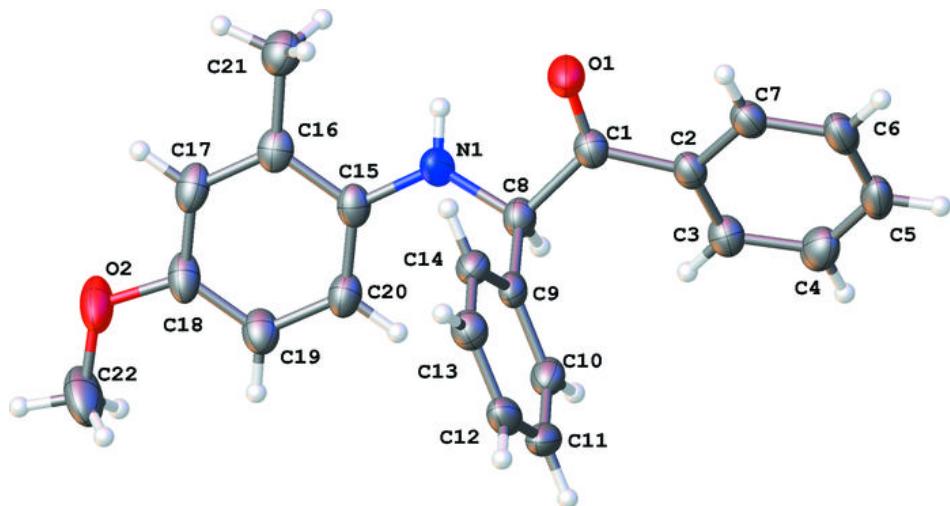
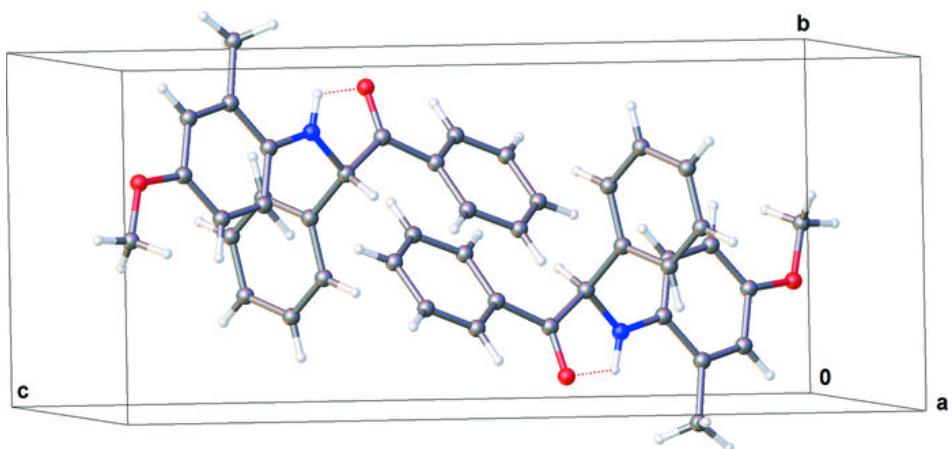


Fig. 2



## supplementary materials

---

Fig. 3

